***Supplementary Material***

Degradation of tris(1-chloro-2-propanyl) phosphate by the synergistic effect of persulfate and zero-valent iron during a mechanochemical process

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**1. Materials and method**

## 1.1. Sample extraction

In order to detect the degradation rate and intermediate of TCPP. 0.05 g of each milling sample was dissolved in 10 mL of ethyl acetate, and then vortexed for 2 min, following by ultrasonic treatment for 15 min. The supernatant was collected after centrifugation at 3000 rpm for 5 min. The above steps were repeated three times and all the supernatant was collected. The supernatant was concentrated to 1mL by a rotary evaporators (Rotavapor R-200, Buchi, Switzerland), and then the concentrated solution was filtered by a 0.22 μm polyether sulfone (PES) filter before determination by GC/MS and LC-QTOF-MS instrumental analysis.

**Table S1. The FT−IR characteristic peaks of relevant functional group**

|  |  |
| --- | --- |
| Functional group | Absorption peak/cm−1 |
| O-H | 3262 |
| C=O | 1642 |
| CH2 | 1428 |
| C-C | 1281 |
| P-O | 1110 |
| P-O-C | 1049 |
| C-Cl | 695 |

**Table S2 Characteristics of three types of soils.**

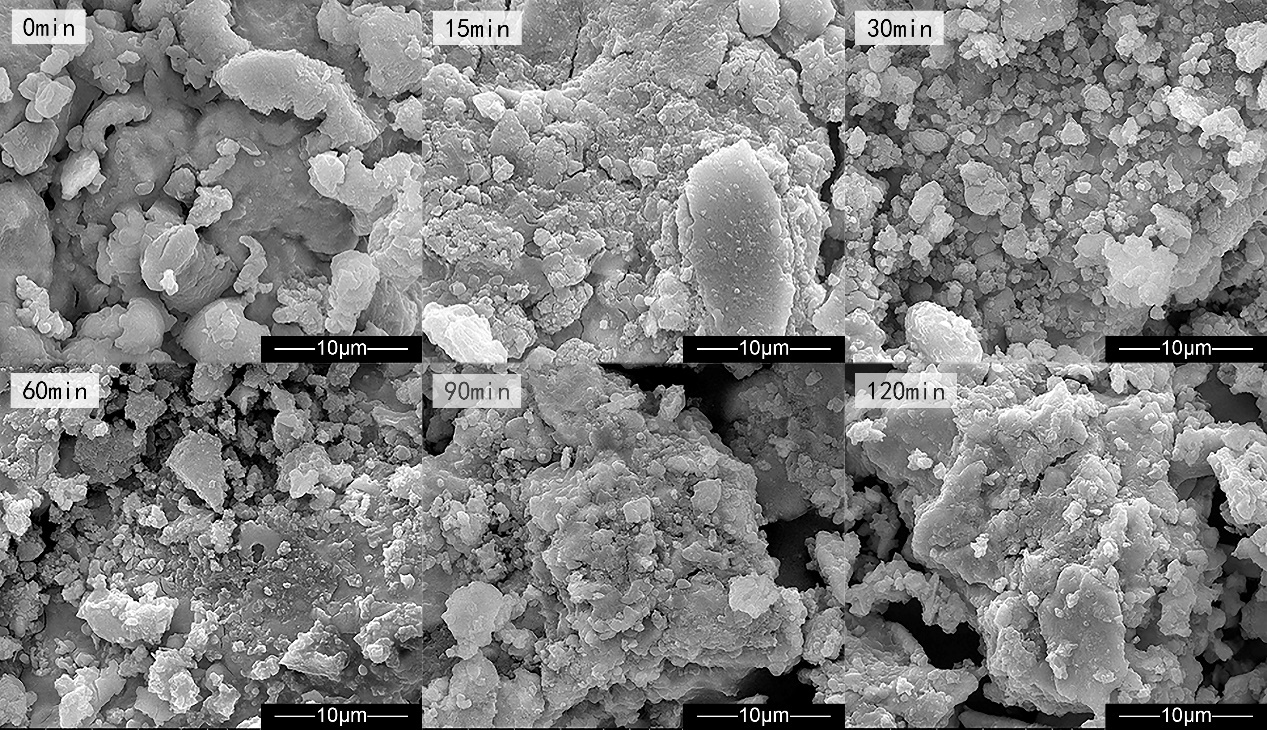
|  |  |  |  |
| --- | --- | --- | --- |
| Soil types | pH | Fe (mg/L) | SOM (g/kg) |
| Red soil | 4.98 | 166.34 | 7.996 |
| Yellow soil | 5.46 | 114.76 | 7.352 |
| Black soil | 5.72 | 125 | 18.376 |



## Fig.S1. GC/MS chromatogram of 10mg/L TCPP standard



**Fig. S2 Standard curve of the concentration of two isomers of TCPP**



**Fig. S3 SED analysis of the milled samples**

**Fig. S4 Particle size distribution of samples (measured by laser particle size analyzer, Dandong Baite, BT-9300ST, China)**

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**Fig. S5 Color change of ball milled samples**

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**Fig. S6. The concentrations of the produced Fe2+ and Fe3+ in MC process**

**Fig. S7 The Mass spectrum of the TCPP degradation intermediates in the PS-Fe-MC progress**

## Table S1 The names and structural formulas of TCPP isomers tested in the experiment

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Abbreviation | Common name | | Structure | | CAS number |
| TCPP 1 | | Tris(2-chloro-1-methylethyl) phosphate |  | 13674-84-5 | |
| TCPP 2 | | Bis(3-chloro-1-propyl) (2-chloro-1-methylethyl) phosphate |  | 137888-35-8 | |

## Table S2 Deconvolution parameters of the Fe 2p spectra

|  |  |
| --- | --- |
| Name | Binding energy position (eV) |
| ZVI 2p 3/2 | 706.7 |
| ZVI 2p 1/2 | 720.0 |
| Fe(Ⅱ) 2p 3/2 | 712.7 |
| Fe(Ⅱ) 2p 1/2 | 723.8 |
| Fe(Ⅱ)sat 2p 3/2 | 714.7 |
| Fe(Ⅱ)sat 2p 1/2 | 726.9 |
| Fe(Ⅲ) 2p 3/2 | 710.9 |
| Fe(Ⅲ) 2p 1/2 | 725.1 |
| Fe(Ⅲ)sat 2p 3/2 | 716.9 |
| Fe(Ⅲ)sat 2p 1/2 | 728.8 |

## Table S3 Deconvolution parameters of the Cl 2p spectra

|  |  |
| --- | --- |
| Name | Binding energy position (eV) |
| C-Cl 2p 3/2 | 200.7 |
| C-Cl 2p 1/2 | 202.5 |
| Cl- 2p 3/2 | 198.2 |
| Cl- 2p 1/2 | 200.0 |

## Table S4 TCPP degradation intermediates in the PS-Fe-MC progress

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| **Products label** | **Molecular formula** | | **［M+H］+** | **∆ppm** | **Retention time(min)** |
| **A** | | **C6H13Cl2O4P** | **251.0003** | **0.6** | **15.23** |
| **B** | | **C3H8ClO4P** | **174.9922** | **0** | **15.2** |
| **C** | | **C9H17Cl2O5P** | **307.0275** | **3.8** | **10.43** |
| **D** | | **C9H17Cl2O6P** | **323.0273** | **0.2** | **9.74** |
| **E** | | **C6H12ClO6P** | **247.0013** | **0.7** | **12.51** |